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THE ANALYSIS OF STEEL-WORKS MATERIALS

By HARRY BREARLEY
AND
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LONGMANS, GREEN, AND CO.
LONDON, NEW YORK AND BOMBAY

THE
ANALYTICAL CHEMISTRY OF URANIUM

BY

HARRY BREARLEY

JOINT AUTHOR OF "THE ANALYSIS OF STEEL-WORKS MATERIALS"

LONGMANS, GREEN, AND CO.
39 PATERNOSTER ROW, LONDON
NEW YORK AND BOMBAY
1903

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PREFACE

ANY ONE desirous of mastering the practical determination of any metal in its ores and in the commercial products whose value it influences, perhaps could do no better, if immediate results were of no consequence, than to choose a gravimetric and a volumetric process which worked well with pure solutions of the metal's salts, and then to set about observing the influence exerted by variations in the mode of operating and by the presence of other metallic salts. Such a procedure, at any rate, usually gives ultimate satisfaction and confidence, and makes valuable additions to one's permanent knowledge of chemical facts, as well as, now and again, eliminates some fictions.

Some years ago the author was invited to familiarize himself with the assay of some metals of which he had no special knowledge with the view of taking charge of an analytical practice in which these metals were to be largely dealt with. As far as time permitted, his mode of doing so was that just indicated. In a few instances the outlines then accumulated have been filled in, and an orderly arrangement of the material made, so that it might be as useful as possible in the laboratory.

If each element were dealt with in such a manner, the accumulated result would save much vexation and laborious search. This, in time, some industrious person, free from the necessity of earning a living, may do. Meanwhile this booklet, by the manner in which it is received, may assist one to decide whether more of a like kind are desirable, or whether the author is mistaken in the general usefulness of the plan.

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THE ANALYTICAL CHEMISTRY OF URANIUM

INTRODUCTORY NOTE.

Uranium was discovered by Klaproth in 1789, but the actual metal was isolated by Peligot in 1842; what had previously been regarded as the metal was really its lower oxide (UO_2).

The ore occurs in Saxony, Cornwall, and America, but the deposits are generally of a very sparing character. The Cornish mine at St. Stephen's, where ore (mostly uranic phosphate) is said to occur in a continuous lode from 3 to 5 feet thick, is exceptional.

Uranium is closely related (chemically) with chromium, molybdenum, and tungsten. These latter metals have each an acid-forming trioxide; the trioxide of uranium, however, acts both as an acid (sodium uranate) and a base (uranyl nitrate). Early papers on the chemistry of uranium are unintelligible unless one bears in mind, not only that the protoxide was mistaken for the metal, but also that the atomic weight of the metal has been doubled in recent times.

Uranous oxide is formed by heating U_3O_8 in hydrogen. It is the base from which the unstable uranous salts are derived, *e.g.* $U(SO_4)_2$.

Uranic oxide unites with strong bases to form uranates which have a composition analogous to dichromates, *e.g.* $Na_2U_2O_7$ (sodium uranate). But it also acts as a base in the formation of uranyl salts ; *e.g.* UO_2SO_4 and $UO_2(NO_3)_2$.

Uranoso-uranic oxide occurs in nature as pitchblende, and may be prepared artificially by heating any oxide in air or oxygen. When digested with nitric acid it dissolves to $UO_2(NO_3)_2$, and this salt by limited ignition is converted to the trioxide, of which it is a derivative. When U_3O_8 is very strongly ignited it loses oxygen, and passes to U_2O_5 ; this, it is surmised, is the change which takes place — with the formation of an intense black colour — when porcelain painted with the uranic glaze is fired.

Higher oxides, which give rise to perurananates, are formed by adding hydrogen peroxide to uranyl solutions. These have been investigated by Fairley and others, and made use of to a limited extent in the analysis of uranium compounds.

I.

MODES OF DETERMINING URANIUM.

Gravimetrically, the determination of uranium depends on its precipitation as alkaline uranate, which is weighed as such or ignited to oxide ; as protoxide by means of ammonium sulphide ; or as phosphate.

The yellow precipitate formed when caustic soda is added to a solution of uranyl nitrate is a hydrated diuranate, which loses water merely on ignition, and may be weighed as $Na_2U_2O_7$. When ammonia is used instead of soda, the analogous precipitate is decomposed on ignition, and yields a compound approximating more or less to U_3O_8, according to circumstances. If only moderately heated the ammonium uranate does not ignite uniformly to the dark green colour of U_3O_8 ; it contains visible streaks and patches of a yellow or brown colour.[1] At higher temperatures, however, the oxide becomes practically black (though it gives a green streak), and loses slightly in weight. According to Zimmermann, pure U_3O_8 is formed only when heated and cooled in a current of oxygen; if heated in air and cooled quickly a small amount of oxygen is lost, and in an indifferent gas the loss is still greater: nevertheless, the elaboration of a current of oxygen may be omitted in technical assays, as the error is comparatively trifling.

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[1] These are said to be formed when traces only of alkaline earths are present.

The precipitation of uranium by means of ammonium sulphide is accurate, and lends itself to a number of important separations; but great care must be taken to exclude carbonates from the solution and the reagent, as they prevent a complete precipitation. The precipitate thus formed may be ignited in hydrogen and weighed as UO_2, or ignited in air to U_3O_8. Kern found that $(NH_4)_2U_2O_7$ could be readily converted to UO_2 by ignition in hydrogen, but that a previously ignited precipitate could not be so reduced and reweighed for purposes of control, particularly if the operation was carried on in a porcelain crucible.

The precipitate of UO_2HPO_4, which is formed when sodium phosphate is added to an acetic acid solution containing uranium, is gelatinous, and washes badly—worse than ammonium uranate even,—and is to be desired only on account of the lower factor needed to convert the weight of the ignited precipitate to metallic uranium. This difficulty can be partly met, as Kern points out, by the careful use of dihydrogen ammonium phosphate instead of the sodium salt; the most satisfactory procedure, however, is that noticed in the next section.

Attempts to weigh the dried precipitate as ammonium uranyl phosphate ($UO_2NH_4PO_4$) have not met with much success; it is customary, therefore, to ignite it apart from the paper, and weigh as pyrophosphate $(UO_2)_2P_2O_7$. If ignited at low redness the precipitate has a pure yellow colour, but at higher temperatures it is partly reduced and becomes green. It is feasible, of course, to ignite precipitate and paper together, and then treat the green residue with nitric acid, evaporate, and ignite to low redness, so as to restore the characteristic yellow colour.

Uranium may be electro-deposited from acetate solutions, and ignited to U_3O_8. Separations from barium, calcium, magnesium, and zinc have been based on this reaction, but it is interfered with by many of the common metals, particularly those of the iron group.

Volumetric processes for the determination of uranium have attracted little attention, and failed to inspire much confidence. The titration with a standard solution of microcosmic salt, using ferrocyanide as a spot indicator, is known rather as the inverse of a common way of titrating phosphoric acid than as a widely usable means of estimating uranium. Guyard's process depends on the precipitation of a triple ammonium-uranium-manganese phosphate when a solution of manganic metaphosphate is added to an acetate solution of uranium. The process is only suitable for the estimation of large amounts of uranium.

The volumetric method most in vogue depends on the reduction of UO_3 to UO_2 in acid solutions by means of zinc, aluminium, or magnesium, followed by titration with permanganate in the usual manner, contact with air being prevented as much as possible. There are no decisive colour changes to indicate complete reduction, so that an abundance of the metallic reducing agent must be used, and a time limit, depending on the amount of uranium dealt with, must be adhered to. Under the most favourable circumstances this limit exceeds the time needed to perform a gravimetric estimation, so that the usefulness of volumetric processes generally are confined to special cases.

II.

THE DETERMINATION OF URANIUM AS PHOSPHATE.

About ten times as much microcosmic salt as there is judged to be uranium present is added to the boiling solution; then dilute ammonia, until a small precipitate is formed; then just as much nitric acid as clears the solution, but no more; and, finally, sodium thiosulphate equivalent to about 10 grams of the crystallized salt. The solution becomes intensely yellow, and almost immediately deposits a voluminous precipitate. After boiling for ten or fifteen minutes, this precipitate contains all the uranium, and is so dense that it settles immediately, and may be easily washed by decantation and collected on the filter. The precipitate is transferred to a porcelain crucible, ignited, the green residue weighed, just moistened with nitric acid and gently heated. It dissolves to a yellow solution with slight effervescence, and is then dried, ignited at low redness, and the yellow residue weighed. $((UO_2)_2P_2O_7 \times 0{\cdot}6681 = U.)$ Pure pyrophosphate dissolves entirely when heated with nitric acid; in practice an insoluble residue, weighing a milligram or so, may be found.

If a series of weighings of the green and yellow residues be scrutinized, a fairly uniform relation is seen to exist between them, and, for many technical purposes, it is unnecessary to take the extra trouble of converting the former to the latter. This is illustrated by the following table, which also justifies the above mode of operating. In each case the solution was diluted to about 300 c.c. before precipitating.

U. taken.	Green Residue.		Yellow Residue.	
	Weighed.	× 0·6855.	Weighed.	× 0.6681.
0·2220	0·3233	0·2216	0·3322	0·2219
0·1226	—	—	0·1840	0·1229
0·1110	0·1617	0·1108	0·1663	0·1111
0·0666	0·0972	0·0666	—	—
0·0444	0·0660	0·0452	0·0675	0·0448
0·0220	0·0320	0·0219	0·0340	0·0227
0·0111	0·0170	0·0116	0·0170	0·0114

The green precipitate is decidedly less hygroscopic than the yellow pyrophosphate, and may be easily brushed from the crucible even after strong ignition: it, however, is too hygroscopic to make the practice of brushing from the crucible a commendable one. Fresenius' statement, that the yellow salt is not hygroscopic, is contrary to all my experience; in fact, a good dessicator and quick weighing are imperative if its weight is to be depended on. The residue disintegrates and leaves the sides of the crucible after absorbing moisture from the air.

In each of the following experiments 30 c.c. of a standard solution of uranyl nitrate, containing 0·2045 gram uranium, was used, and the volume of the solution precipitated was about 300 c.c. The figures in brackets represent the amount of uranium obtained when calculated from the green residue by the factor already used (0·6855.) The agreement of the two sets of figures confirms the statement already made, that its composition is sufficiently constant to warrant its weight being used in calculating the percentage of uranium for technical purposes.

Acetic Acid.—The operation is in no way affected by moderate amounts of this acid.

Acetic acid (c.c.)	0	10	30	50
U obtained (gr.)	0·2045	0·2042	0·2047	0·2049
Green precipitate		(0·2046)	(0·2053)	(0·2048)

Time of Boiling.—When large amounts of uranium are being handled, it is sometimes a difficult and dangerous operation to boil the solution from ten to fifteen minutes on account of the vigorous bumping. Nearly all the uranium is down after boiling for a minute or two, but this can hardly be depended on. The danger may be avoided after adding the thiosulphate, by heating only as long as is necessary to liberate a considerable amount of sulphur and form a precipitate, which settles readily. Then, if 5 c.c. strong ammonium acetate are added to destroy any free mineral acid and complete the precipitation, little or no further boiling is necessary. A small amount of acetate may be added after long boiling, if further assurance of a complete precipitation is desired. The added acetate is harmless, as the following tests show :—

Acetate (c.c.)	5	30	50
U obtained (gr.)	0·2046	0·2045 (0·2051)	0·2044 (0·2049)

Ammonium Nitrate (20 grams).

0·2048 (0·2050) instead of 0·2045 gram uranium.

Ammonium Chloride (20 grams).

0·2042 (0·2046) instead of 0·2045 gram uranium.

Ammonium Sulphate (20 grams).

0·2038 (0·2046) instead of 0·2045 gram uranium.

The precipitated uranyl phosphate, formed on neutralizing, clears up less readily on adding nitric acid than when ammonium sulphate is absent. The same observation applies to the added sodium sulphate.

Sodium Nitrate (20 grams).

0·2047 (0·2043) instead of 0·2045.

Sodium Chloride (20 grams).

0·2049 (0·2043) instead of 0·2045.

Sodium Sulphate (20 grams).

0·2035 (0·2036) instead of 0·2045.

Potassium Nitrate (20 grams).

Potassium salts are carried down. The weight of the ignited pyrophosphate was equivalent to 0·2200 gram uranium, but, on dissolving and reprecipitating a residue corresponding to the correct weight of uranium is obtained, *i.e.* 0·2040 gram.

Microcosmic Salt.—Varying excesses of this reagent may be used without detriment, but more than the theoretical amount is necessary. For example, the author, when using 5 grams instead of the usual 2 grams, of microcosmic salt, obtained a precipitate having a weight equivalent to 0·2042 gram uranium. On dissolving this and reprecipitating, without adding any more microcosmic salt, the resulting precipitate was equivalent to 0·1988 gram; on again dissolving, adding microcosmic salt, and again precipitating, the weight of the residue was equivalent to 0·2030 gram. This shows that the second precipitate, which contained nearly all the uranium, was a basic compound.

Properties of the Precipitate. — The ignited pyrophosphate is more readily soluble in nitric than in sulphuric or hydrochloric acid; contamination with ferric oxide greatly decreases the solubility. It is easily attacked by fused sodium carbonate or caustic soda, but on extracting with water the yellow residue contains only about 90 per cent. of the uranium operated with. The portion in the filtrate is at once detected by the yellow colour which carbonated alkali solutions give with hydrogen peroxide.

III.

THE SEPARATION OF URANIUM.

SEPARATIONS FROM IRON.

By Acetates.—The separation of uranium from iron, by precipitating the latter as basic acetate, was referred to in a general way by Gibbs,[1] but no particulars were given. Some phases of the process were studied by Rheineck,[2] who suggested that the operation was a delicate one, and observed that a crystalline precipitate is formed at once in concentrated, and, after some time, in more dilute solutions, when sodium acetate is added to any salt of the oxide of uranium.

The following experiments were made with the object of determining in which direction the weakness of the separation lay, rather than with the idea of improving it for regular service. Five grams of bar iron were dissolved in hydrochloric, and oxidized with nitric acid, 25 grams ammonium chloride and 150 c.c. standard uranium solution added, and the mixture then neutralized and divided into five equal portions.

---

[1] *Chemical News*, xi. 102.

[2] *Chemical News*, xxiii. 233.

(*a*) Was diluted to 300 c.c., and boiled. The iron was almost entirely precipitated, and no acetate whatever was added: 0·2055 gram uranium was found in the filtrate, instead of 0·2045 gram.

(*b*) Five cubic centimetres strong ammonium acetate were added, which precipitated the iron immediately in the cold. The solution was then boiled three or four minutes. The filtrate contained only 0·0853 gram uranium.

(*c*) Treated like (*b*), but 10 c.c. acetic acid were added before the acetate. A precipitate formed after very little heating. The filtrate contained 0·1994 gram uranium.

(*d*) Treated as (*a*), but added 0·5 c.c. acetate to the boiling solution, to completely precipitate the iron—0·2000 gram uranium in the filtrate.

(*e*) Treated as (*a*), but added 5 c.c. acetic acid to the cold solution, and 1 c.c. ammonium acetate to the boiling solution. The precipitation of the iron was complete; in fact, less acetate might have been used. The filtrate contained 0·2048 gram uranium.

There is no doubt that when phosphoric acid is absent, and the proportion of uranium is large enough to yield a sufficient amount in the filtrate, without having to deal with unwieldy precipitates of basic ferric acetate, this separation is a reliable one. The neutralized solution, however, should contain 1 or 2 per cent. of free acetic acid, and no more acetate should be used than will comfortably precipitate all the iron. With these precautions the separation appears to be quite as easy as that of iron and nickel by the same process. It is probable, also, that others of the alkaline salts which have the power of precipitating neutralized ferric solutions might advantageously be substituted for ammonium acetate.

By Alkalis.—When ammonium carbonate is added to a solution containing iron and uranium, the former is precipitated, but may or may not be free from the latter. A separation, which is practically perfect, can be made by pouring a solution containing as much as 2 grams of iron into a solution, which is kept agitated, of ammonium or sodium carbonate or sodium bicarbonate. A little iron, however, is apt to remain in solution, and it is customary, if its presence is objectionable, to allow it to settle out after prolonged standing or to precipitate it with ammonium sulphide.

Separations made in this way gave the following results :—[1]

	Fe present.	U present.	U found.
Am. carb.	2·0 grams	0·0621	0·0623
Sod. carb.	2·0 „	0·0621	0·0622
Sod. bicarb.	2·0 „	0·0621	0·0619

Caustic alkalis, of course, precipitate the uranium along with the iron, but, according to Walker, it remains in solution if hydrogen peroxide is also present.[2] This separation appears to be quite successful only when the proportion of iron is not large and the total weight of the metals dealt with is small.

By Ether, etc.—Kern's valuable contribution to the quantitative separation and determination of uranium[3] deals particularly with the separation of iron. His experimental work is confined to the ether-extraction process, which in other directions is already well known, and he finds that the most complete separations are obtained by using hydrochloric acid of 1·10 specific gravity. More than one extraction is necessary, and it is doubtful whether the process is a desirable one unless ether extractions in other directions are being regularly carried out.

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[1] I am indebted to my friend, Fred Ibbotson, B.Sc., for these results.

[2] *Jour. Amer. Chem. Soc.*, xx. 513.

[3] *Jour. Amer. Chem. Soc.*, xxiii., and *Chemical News*, vol. lxxxiv.

A separation described by Rose depends on the precipitation of the two oxides by ammonia, ignition in a stream of hydrogen, and extraction of the metallic iron with hydrochloric acid. Or the iron may be volatilized from the mixture by heating it in an atmosphere of the gaseous acid.

The electro-deposition of iron from oxalate solutions is said to lead to a perfect separation.

The separation by means of alkaline acetates is trustworthy and convenient within the limitations already referred to. Its value and accuracy is further attested by the following results; nevertheless, some other alkaline salt instead of acetates could probably be used with advantage:—

Fe present.	°/₀ U present.	°/₀ U found.
5 grams	0·817	0·836
3 "	3·18	3·15
2 "	4·73	4·77
1 "	20·45	20·40
2 "	10·22	10·15

When a minor constituent of any mixture is required to be estimated, it is generally best to separate it by direct precipitation. This can be accomplished with a mixture of uranium and iron by applying, under suitable circumstances, the method already described for determining uranium in pure solutions. The operations are similar to those used for separating small amounts of aluminium and chromium from steel as phosphate. The separation can, of course, be used equally well when the proportion of uranium is great.

The iron being in the ferrous state, and preferably as chloride, heat the solution nearly to boiling, add microcosmic salt in the proportion already indicated, and then dilute ammonia until the precipitated flocks of ferrous hydrate are no longer quite redissolved. Clear with the smallest possible excess of hydrochloric acid, add 10 grams of sodium thiosulphate and 20 c.c. acetic acid, and boil for ten to fifteen minutes. Allow the precipitate to settle, filter as quickly as possible, and wash with hot water containing a few cubic centimetres per litre of acetic acid and ammonium acetate.

The residue, after redissolving from the filter and the accompanying sulphur, may be reprecipitated to separate the remaining portion of iron, which, when the operation is properly performed, is rarely greater and often much less than the amount of uranium. Or, instead, the residue may be ignited, fused with sodium carbonate, dissolved in hydrochloric acid, filtered after evaporation to remove any such insoluble body as silica, and reprecipitated as before. The ignited precipitate has the characteristic green colour, and may, if desired, be converted to the yellow pyrophosphate. Not more than a negligible trace of ferric oxide, carried down by the sulphur in the final precipitation, should be present. The following results were obtained in this way:—

Iron Used.	% U present.	% U found.
5 grams	0·136	0·136
5 "	0·273	0·280
5 "	0·546	0·555
10 "	0·068	0·070
5 "	0·819	0·836

The addition of ammonium acetate to complete the precipitation and shorten the boiling is not commendable when much iron has to be separated. In order to avoid violent bumping, I have found the following plan to work well: Slide a small widely perforated filter-plate into the solution, and limit its movement along the bottom of the beaker by putting the drawn-out end of a glass stirring-rod through one of the holes. A similar plate, allowed to move freely, breaks the steam envelopes in a haphazard way, and may cause the liquid to boil over.

SEPARATIONS FROM NICKEL, COBALT, AND ZINC.

These metals are usually separated from uranium by passing a stream of sulphuretted hydrogen through a boiling solution of the acetates (Gibbs). Zinc only may be separated by means of ammonium carbonate (Rammelsberg). Nickel and cobalt, on the other hand, remain in solution when an emulsion of barium carbonate is added to a mixture of the metals containing a small amount of free acid and some ammonium chloride. Uranium is precipitated, but must, subsequently, be separated from the excess of barium carbonate. The electro-deposition of the uranium as hydrated oxide from an acetate solution is not interfered with by the presence of zinc.

The presence of either nickel, cobalt, or zinc in no way interferes with the estimation of uranium by the method already given for pure solutions. The following are results of test analyses :—

Metal present. (0·10 grams.)	Acetic Acid (c.c.).	U present.	U found.
Zinc . . .	0	0·2045	0·2056
Zinc . . .	50	0·2045	0·2056
Nickel . .	0	0·2045	0·2053
Nickel . .	50	0·2045	0·2049
Cobalt . .	0	0·2045	0·2046
Cobalt . .	50	0·2045	0·2049

The respective filtrates can be used conveniently for determining the separated metal; those containing zinc or cobalt (after boiling with nitric acid to destroy the thiosulphate) by precipitating as the double ammonium phosphate, and those containing nickel by making alkaline and titrating with potassium cyanide and silver iodide.

SEPARATIONS FROM MANGANESE.

The separation of manganese from uranium does not appear to have been much studied. It can be separated, according to Rammelsberg, with ammonium carbonate alone, but the separation generally described is that applicable to other members of its group, viz. precipitation with sulphuretted hydrogen from a solution containing alkaline carbonates.

When uranium is precipitated as phosphate in the manner described above, it carries down a small portion of any manganese present in the solution. A series of separations of 0·10 gram Mn from 0·2045 gram uranium were made in solutions containing amounts of acetic acid, varying from 0 to 50 c.c. The manganese associated with the uranyl pyrophosphate varied from 0·0013 to 0·0006 gram.[1] When the $Mn_2P_2O_7$ equivalents of these figures were deducted from the total weights, the remainders calculated to amounts of uranium varying between 0·2030 and 0·2050 gram.

SEPARATIONS FROM ALKALIS AND ALKALINE EARTHS.

The separation of alkalis from uranium is of technical importance, as potash is a very considerable constituent of an industrial ore—carnotite. Alkalis, alkaline earths, and magnesia can be separated by precipitating the uranium several times with ammonia in the presence of ammonium chloride; but, considering how badly ammonium uranate filters, this separation is an undesirable one. It is much better to precipitate the uranium from acetic solutions with ammonium phosphate, which, according to Fresenius, effects a complete separation from alkalis and alkaline earths. Potash is the most difficult element to separate, and if present in large amounts a second precipitation may be necessary.

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[1] The amount is easily determined by dissolving in nitric acid oxidising with sodium bismuthate, and titrating the permanganate formed with ferrous sulphate.

The separation of calcium as oxalate from ammonium carbonate solutions has been strongly disparaged by Alibigoff. He effects the separation—also from alkalis and strontium, but not barium — by boiling the solution, containing ammonium chloride or nitrate, with an emulsion of mercuric oxide.

The separation of barium with sulphuric acid is accurate and convenient. The separation of calcium and strontium, in the same way, from alcoholic solutions is practised, but for small amounts is much less satisfactory.

The precipitation of uranium as phosphate by boiling acetic acid solutions with thiosulphate effects a complete separation from calcium. If no acetic acid is present a portion of the calcium is precipitated as phosphate. The following tests were made on solutions (300 c.c.) containing 0·10 gram of calcium and 0·2045 gram uranium :—

Acetic Acid (c.c.).	U found.
0	0·2536
5	0·2063
10	0 2056
20	0·2047
50	0·2043

The lime in the filtrate can be determined simply by adding an excess of ammonium oxalate, allowing the solution to cool, filtering, igniting the residual oxide, transforming it to sulphate, and weighing. The weight of the oxide cannot be relied upon, as it may contain sulphuric acid.

Magnesium does not interfere with the precipitation of uranium.

Acetic acid (c.c.)	.	0	50
U obtained (gr.)	.	0·2046	0·2051

SEPARATION FROM COPPER AND OTHER METALS PRECIPITATED BY SULPHURETTED HYDROGEN.

The presence of metals whose sulphides are insoluble in very dilute acid solutions would interfere with the chosen method of precipitating uranium. When any single member of this group—which includes lead, cadmium, mercury, bismuth, copper, silver, tin, antimony, and arsenic

—has to be separated, its precipitation with sulphuretted hydrogen, from a suitably acidified solution, can be arranged. But if it is desired to detect and separate any (unknown) one of them, the acidity must be slight enough to permit the complete precipitation of the one whose sulphide is most easily dissociated, viz. lead. A complete precipitation of lead as sulphide can be made from solutions containing 2 per cent. of concentrated hydrochloric acid. When 2½ per cent. of concentrated acid is present the precipitation is incomplete. This is the conclusion arrived at by Kern, after making a series of quantitative separations under varying conditions of acidity and temperature.

SEPARATION FROM SILICA.

The ignited pyrophosphate always contains silica when the precipitation has been made from a solution containing a soluble silicate. Thus, from a solution containing sodium silicate equal to 0·184 gram silica, 0·2045 gram uranium carried down in the two cases 0·010 and 0·006 gram silica respectively.

If the silica is not removed by evaporation with acids prior to the precipitation, it remains insoluble on digesting the pyrophosphate with nitric acid, and can be filtered off, weighed, and approximately allowed for, or the uranium may be reprecipitated from the filtrate.

Uranyl pyrophosphate loses weight when treated with hydrofluoric acid alone. With a mixture of hydrofluoric and nitric acid no loss occurs; but it is necessary to evaporate a second time with nitric acid only, in order to secure the yellow pyrophosphate free from fluorides. A mixture of hydrofluoric and sulphuric acids may also be used if the weight of the green residue is being depended upon; but in that case a very strong ignition is needed in order to drive off all the sulphuric acid.

SEPARATION FROM MOLYBDENUM.

Molybdenum is partly precipitated as sulphide when an acid solution containing it is boiled with thiosulphate. The means of precipitating uranium previously adhered to cannot therefore be used in the presence of this element; but a very satisfactory determination can be made by raising the clear acid solution of the two metals to boiling point, and adding at once the required amount of phosphate mixed with as much hot ammonium acetate as will destroy all the free mineral acid.

The precipitate is then washed well by decantation, and collected on the filter. The washing is somewhat facilitated by adding a small quantity of floured sulphur during the decantation.

Mo present .	0·0000	0·1000	0·2000	gram
U obtained .	0·2045	0·2046	0·2046	,,

The estimation of the molybdenum in the filtrate, at least so far as its precipitation as lead molybdate is concerned, is complicated by the presence of phosphoric acid; it may therefore be remarked, that molybdenum can be estimated on a separate portion in the following manner, without previously removing the uranium:—

To the hot solution containing a few cubic centimetres hydrochloric acid in excess, add enough lead acetate to combine with all the molybdenum present, and then sufficient ammonium acetate to destroy the free hydrochloric acid. The precipitated lead molybdate, which is very granular and easily washed, is ignited and weighed. The traces of uranium which the precipitate may contain can be recovered, if desired, by pouring the hydrochloric acid solution of it into caustic soda; the error which they introduce is, however, quite insignificant. The uranium in the original filtrate may be estimated as phosphate only after separating the lead either by passing sulphuretted hydrogen or evaporating with sulphuric acid.

SEPARATION FROM TUNGSTEN.

Tungsten may be completely precipitated from neutral solutions by uranyl nitrate or chloride (but not acetate, according to Hitchcock) in the form of uranyl tungstate. This fact, and the marked tendency of tungstic acid to precipitate spontaneously from acidified solutions, prevent uranium from being determined as phosphate in a mixture of the two metals. Evaporation to dryness on the water-bath with a good excess of hydrochloric acid is a very satisfactory means of making the separation, because, after boiling with dilute hydrochloric acid (1 in 5), all the tungsten is left in the residue, and requires to be merely ignited and weighed as tungstic oxide; and all the uranium is in the filtrate ready to be estimated in the usual manner. From a synthetic mixture of 0·1992 gram tungsten and 0·2045 gram uranium there were obtained 0·1989 and 0·2040 gram respectively.

SEPARATION FROM CHROMIUM.

The separation from this element can be made most easily when it exists as chromic acid; as chromic oxide it is completely precipitated along with the uranyl phosphate. The chromium can be conveniently oxidized with sodium peroxide, but it is almost impossible in the presence of uranium to entirely decompose the excess of peroxide by boiling. It is therefore necessary to proceed as follows:—

Neutralize most of the free acid with sodium carbonate, and then add sodium peroxide until the solution becomes strongly alkaline. Both metals are precipitated and again dissolved, the chromium being entirely in the higher state of oxidation. Boil the solution well, then acidify, and add a small volume of decinormal permanganate (10 c.c. should be ample), and boil for about ten minutes. If the permanganate decomposes at all (thus showing that a portion of the chromic acid was reduced by the residual hydrogen peroxide in the acid solution) then filter off the manganic oxide through asbestos fibre, and destroy any unused permanganate with a few drops of hydrochloric acid. The acid liquor is now made slightly alkaline with ammonia, decidedly acid with acetic, and when it boils briskly an excess of microcosmic salt is added, and the filtration proceeded with as in the separation from molybdenum. The chromium in the filtrate is estimated volumetrically.

Uranium could probably be precipitated from an acetic solution of chromic acid as uranyl vanadate; the converse of this is Klecki's method for separating vanadium from chromium. The uranium may also be precipitated with caustic soda, or the chromium may be thrown down with mercurous nitrate. Other means of making the separation are those of electro-deposition, and that depending on ignition of the oxides in a current of hydrogen and extraction of the uranium with nitric acid.

SEPARATION FROM VANADIUM.

Vanadium is an essential constituent of Carnotite, and occurs also in small amounts in other uranium ores. Artificial compounds of uranium and vanadium do not occur in commerce, and therefore the separations to be found in the literature of the subject refer almost exclusively to the analysis of particular ores, and may not be generally applied. For instance, such amounts of vanadium as occur in pitchblende are separated (Lallemand) by pouring the filtered nitric acid solution of the mineral into sodium carbonate, which causes the vanadium to go down with the iron and the uranium to pass into solution. Fritchle, on the other hand, precipitates the dissolved carnotite with a mixture of sodic hydrate and carbonate, which throws out iron and uranium, and leaves vanadium in solution. The separation with mercurous nitrate and mercuric oxide (Langmuir), and the extraction of uranium with ammonium nitrate after evaporating the mixture to dryness with nitric acid are both more widely useful.

The analysis of a complex ore may often be completed without any separation of uranium and vanadium being made. This is accomplished by determining vanadium volumetrically with ferrous sulphate and permanganate, or colorimetrically with hydrogen peroxide, according to its amount, and making the necessary correction in the weight of the precipitated compound used for the estimation of the uranium. The varied uses of devices of this kind cannot be explicitly stated, but it may generally be assumed that, when precipitated with ammonia, the uranium will carry down all the vanadium, and the weighed U_3O_8 will be too heavy by the V_2O_5 equivalent of the amount of vanadium already determined. Also, when uranium is precipitated as phosphate in the ordinary manner, any vanadic acid carried down merely replaces its equivalent of phosphoric acid, and the error introduced may be too trifling for consideration, although the exact amount of it can be arrived at by determining the vanadium in the ignited uranyl pyrophosphate.

An accurate separation can be made by pouring a faintly acid and heated solution of the two metals into a heated mixture of microcosmic salt and 5 or 10 c.c. ammonium acetate. The latter solution is kept violently agitated, while the former is added through a funnel with a stem so constricted that only a thin column passes out of it. The precipitate is washed by decantation and weighed as usual; the combined washings are evaporated to a convenient bulk, and titrated with ferrous sulphate and permanganate. The following results were obtained :—

V present.	U present.	V found.	U found.
0·0503	0·1552	0·0503	0·1554
0·0615	0·1704	0·0615	0·1700
0·1205	0·2045	0·1208	0·2043

SEPARATION FROM TITANIUM.

Titanium is precipitated entirely with the uranyl phosphate in a form very closely approximating to $TiO_2 . P_2O_5$. It occurs so sparingly in ores that a colorimetric estimation satisfies most requirements. In larger amounts it can be separated by adding 30 to 50 c.c. acetic acid to the hot solution, and then sufficient ammonium acetate to destroy the free mineral acid. A large excess of ammonium acetate is to be avoided. (See "Separation of Iron.")

Titanium is completely precipitated by boiling its acid solution with an excess of thiosulphate in the presence of much free acetic acid; uranium is not at all precipitated under these conditions, providing phosphoric acid is absent, but quantitative separations on this principle from synthetic mixtures have not been attempted. When phosphates are present uranium and titanium must be precipitated together, ignited, fused with sodium carbonate, extracted with water, and the residue (consisting of all the titanium and most of the uranium) dissolved in acid, and separated as above.

SEPARATION FROM ALUMINIUM.

Since the introduction of alumino-thermic reductions, experimental metallurgy often requires rapid assays to be made by such means as are not interfered with by aluminium, and it is therefore to be regretted that one's choice of means of separating this element from uranium are so limited. Of the methods proposed for the separation of iron and uranium only two are applicable to the separation of uranium and aluminium, and these with no great measure of success.

The separation of aluminium as basic acetate is never very satisfactory unless preponderating amounts of iron are also present. Its separation as hydroxide with ammonium carbonate is less successful than usual when applied to a mixture containing uranium. Uranium passes entirely into solution when a hot and slightly acid mixture of the two metals is poured in a thin stream into an agitated solution of ammonium carbonate, but much of the aluminium also goes into solution, and it cannot with certainty be assumed that all this will again separate even if allowed to stand twelve or fourteen hours in a warm place, and a small amount of aluminium left unprecipitated has an important influence on the estimation of the uranium on account of its comparatively low combining weight. Boiling, of course, cannot be resorted to, for fear of precipitating uranium.

All the aluminium passes into solution when a mixture of the two metals is poured into aqueous caustic soda, but much uranium also goes into solution, and no amount of boiling causes it to separate again. Uranium is precipitated from acetic acid solutions with nitroso-β-naphthol, and aluminium is not; but persistently reliable results were not obtained with this reagent, which is to be regretted, because the uranyl naphtholate filters and washes much better than most other precipitated compounds of uranium.

Generally in the course of analysis aluminium and uranium are obtained together as phosphates. In this form the separation with ammonium carbonate is much more successful; in fact, it is entirely successful if the phosphates are dissolved in nitric acid, most of the acid neutralized,[1] and the clear heated solution poured into the agitated alkaline liquid. If filtered at once a few milligrams of aluminium phosphate remain in solution, and possibly also traces of uranyl phosphate remain in the precipitate. Standing for an hour or two in a warm place minimizes both these errors.

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[1] A pale yellow crystalline precipitate forms on adding ammonia, and not an amorphous cloud such as when solutions of either aluminium or uranium phosphate are neutralized. The former precipitate does not redissolve so quickly in hydrochloric acid as the latter, and more care must therefore be taken over the neutralization. I do not know the composition of the lemon-coloured crystalline precipitate.

A modification of this separation, which is sometimes to be preferred, is to pour the dissolved metallic phosphates into an excess of sodium carbonate. The clear or merely opalescent liquid is then heated to boiling, but not boiled, removed from the flame, and ammonium chloride (in amount more than equivalent to the excess of sodium carbonate) added. This solution also, if filtered at once, contains a few milligrams of aluminium phosphate; but it is an easy matter to dissolve the uranyl pyrophosphate subsequently obtained (and weighed) in nitric acid, pour the solution into ammonium carbonate, collect the precipitate, and weigh it. The following results were obtained after separating from a gram of aluminium :—

	U present.	U found.
Ammon. Carb.	0·2132	0·2122
Sod. Carb. and Am. Chlor.	0·2132	0·2132

The behaviour of the ignited pyrophosphate when fused with sodium carbonate is referred to on page 12. It might be imagined that a separation of practical utility could be based on this reaction if supplemented by a colorimetric estimation of the small fraction of uranium extracted by digesting with water. Experiment, however, shows that the amount of uranium passing into solution is very much greater when the fused pyrophosphate contains aluminium. But this opportunity may be taken to say that small amounts of uranium, either alone or associated with other metals, may often be conveniently estimated by adding an excess of potassium carbonate and half a gram or so of sodium peroxide, and then matching the colour formed by running a standard uranyl solution into an equal volume of distilled water in which are dissolved similar amounts of potassium carbonate and sodium peroxide. A milligram of uranium can be detected in 60 to 100 c.c. of solution by these means, and the indication is more characteristic than that given with potassium ferrocyanide.

IV.

THE ANALYSIS OF URANIUM ORES AND ALLOYS.

Pitchblende.—Pitchblende is the best-known ore of uranium; it is an impure U_3O_8, containing from 40 to 95 per cent. of that oxide, but not always exactly in the proportion of $UO_2 : 2UO_3$. The following elements have been found associated with it:—sulphur, selenium, phosphorus, calcium, magnesium, aluminium, silicon, vanadium, manganese, arsenic, bismuth, antimony, tin, zinc, lead, iron, cobalt, nickel, copper, and silver. Many of these occur in such small amounts, that their presence is negligible so far as commercial analyses are concerned. Nitrogen and a number of rare earths are also to be found in uraninite from some localities (*vide* "On the Occurrence of Nitrogen in Uraninite, and on the Composition of Uraninite in General," Hillebrand, *U.S. Geological Survey Bulletin*, No. 78, and *Chemical News*, vol. lxiv., p. 221, etc.). The following outline method of analysis meets most industrial demands. The particular form in which the results are expressed is a matter of expediency.

Digest 4 grams of the finely powdered ore with 20 c.c. (1·42) nitric acid, boil down to low bulk, and filter off the siliceous residue. This residue very rarely contains uranium; besides silica, alumina, and ferric oxide, it may contain small amounts of vanadium, tin, and antimony. A further analysis is made, or not, according to circumstances.

The soluble portion is evaporated with hydrochloric acid, diluted, any silica or silver chloride filtered off, and a current of sulphuretted hydrogen passed. The precipitated sulphides of antimony, arsenic, tin, lead, copper, and bismuth, are separated in the usual manner into two groups with sodium sulphide, and the separate determinations made as usual.[1] A separate determination of the arsenic may be desirable, as a portion is probably volatilized from the main portion during the evaporation with hydrochloric acid.

The main filtrate is boiled to expel sulphuretted hydrogen, and nitric acid added to oxidize the iron. It is then made up to 250 c.c.; 100 c.c., or some other volume well suited to the supposed amount of the impurities to be determined, is measured into a flask containing a few crystals of ammonium phosphate, then nearly neutralized, and poured into an excess of aqueous sodium carbonate. The mixture is raised to boiling, and at least as much ammonium chloride added as will destroy the free soda. After standing a few hours the precipitated phosphates are filtered off. Uranium is determined in a portion of the filtrate after acidifying and boiling free from CO_2. The residue is dissolved in sulphuric acid. A portion of this acid solution is reduced with sulphur dioxide, and the iron titrated with permanganate, care being taken to make allowance for any vanadium which may be present. The vanadium may also be estimated, colorimetrically, on this portion if its amount is small compared with the alumina and ferric oxide. The remaining portion of the sulphuric acid solution is boiled with sodium phosphate and thiosulphate to obtain a precipitate from which alumina and titantic oxide may be determined; any uranium not having passed into the ammonic carbonate solution would be found here.

---

[1] It is hardly feasible to give details of these separate determinations, but the general procedure followed by the author is dealt with, in relation to the analysis of white-metal alloys, in "The Analysis of Steelworks Materials" (Longmans, Green & Co.).

To another fraction (100 c.c.) of the main solution, an excess of microcosmic salt is added, then an excess of ammonium acetate to ensure the complete precipitation of uranium, iron, aluminium, etc., from a strongly acetic acid solution. A fraction of this solution is filtered, most of its free acetic acid neutralized, and the lime precipitated as oxalate, and the magnesia subsequently as phosphate. There is generally not sufficient manganese in these ores to interfere with this procedure.

The remaining portion (50 c.c.) of the main filtrate is used for the determination of phosphoric acid *viâ* the molybdate precipitation; or a separate portion may be prepared by digesting the ore with nitric acid in case no appreciable amount of tin is present.

Separate determinations may be made of manganese by digesting 1 gram of the ore with nitric acid, oxidizing the solution with sodium bismuthate, and titrating the permanganate formed; of vanadium, by fusing 1 gram with sodium carbonate, extracting with water, and determining colorimetrically with hydrogen peroxide, or otherwise; of nickel, cobalt, and zinc, by precipitating uranium, aluminium, iron, etc , as phosphates from strongly acetic acid solutions, and applying the usual processes to the filtrate.

The sulphur and moisture are determined as in samples of pyrites.

Carnotite.—This ore, which has attained commercial importance, is found chiefly in Western Colorado. It is a canary yellow compound associated with a fragile sandstone bearing some resemblance to Roscoelite. The analysis is comparatively easy.

Digest 3 grams of the ore with nitric acid, and filter. The ignited siliceous residue may be reported as such, or fused with sodium carbonate, evaporated with hydrochloric acid, etc. Uranium is usually entirely absent from the residue, but vanadium (V_2O_3) and barium sulphate are frequently present along with the usual constituents of silicates.

The filtrate is evaporated with hydrochloric acid, the trifling amount of silica collected, and a few drops of sulphuric acid added to precipitate any barium. If no barium is indicated, sulphuretted hydrogen is passed to separate copper, and, in rare cases, other metals of this group. The filtrate is boiled, oxidized with nitric acid, and made up to 300 c.c. From this point the analysis proceeds on similar lines to that of pitchblende. Vanadium is estimated on a separate portion by titration with ferrous sulphate or otherwise, Carbon dioxide must also be separately determined. The following analyses are of commercial ores :—

PITCHBLENDE.			CARNOTITE.		
	Cornwall.	Joachimsthal.		Insoluble.	Soluble.
U_3O_8	43·47	49·95	UO_3	—	19·22
SiO_2	25·31	18·54	SiO_2	62·85	0·20
Fe_2O_3	0·44	1·14	Fe_2O_3	0·48	0·50
Al_2O_3	2·25	3·25	Al_2O_3	1·09	0·68
MnO	0·20	0·11	CaO	0·17	1·17
CaO	1·34	2·52	MgO	0·18	0·90
MgO	0·25	0·38	BaO	—	—
V_2O_5	0·25	0·02	V_2O_5	0·10	2·90
TiO_2	traces	—	TiO_2	0·09	—
ZnO	—	1·09	P_2O_5	—	traces
P_2O_5	3·28	0·33	SO_3	—	1·03
S	8·25	5·08	CO_2	—	2·70
Ag	0·075	0·03	H_2O	—	3·95
Sn	—	—	K_2O and traces of other metals	—	1·79
Sb	0·62	—			
As	1·00	0·44			
Pb	2·02	1·73			
Bi	0·015	0·24			
Cu	1·32	0·89			
Fe	7·14	8·04			
H_2O	1·50	0·67			
CO_2	—	5·02			
Oxygen combined with Fe not existing as FeS_2, etc.	1·27	0·53			
	100·00	100·00		64·96	35·04

Metal and Alloys.—Metallic uranium was prepared originally by reducing the chloride with potassium, and this process, or some modification of it, was the only one available until a few years ago. Moissan prepared "cast uranium" by submitting an intimate mixture of the oxide and sugar charcoal, contained in a coke crucible, to the electric current. He isolated a crystallized carbide (C_3U_2) whose melting point is much higher than that of platinum, and he prepared samples of the metal containing less than 1 per cent. of impurities. On this latter metal and other samples prepared by the electrolysis of the double uranium-sodium chloride ($UCl_4 . 2NaCl$) he determined some of its properties. Ferro-uranium alloys are formed by the last-mentioned process when iron electrodes are used. The metal and some of its alloys have been prepared by various modified forms of the alumino-thermic process: the author, however, found this method to yield poor and very uncertain results.

Pure uranium is a white non-magnetic metal, and can be easily filed; it does not scratch glass, is easily carbonized, and may be tempered. Its specific gravity is said to be about 18·7, but the carbonized metal usually met with has a specific gravity of about 12 only. When in fine powder the metal burns at a comparatively low temperature (170° in pure oxygen), and also very readily combines with nitrogen. It tarnishes on exposure to air, and also decomposes water to some extent, particularly if heated.

Metallic uranium is decomposed by dilute hydrochloric acid, but black flocks of carbide persist until a few drops of nitric acid have been added. With ferro-uranium alloys or uranium steels it is advisable to filter off this black residue, ignite, fuse it with sodium carbonate, dissolve the melt in hydrochloric acid, and add it to the main solution. The simultaneous precipitation of uranium and separation of iron is made in the manner already described. Carbon in metallic uranium is determined by simple ignition in a stream of oxygen, the resulting carbon dioxide being collected and weighed. The determination of the remaining impurities which affect the value of the metal or alloy for steel-making purposes is made by the usual processes, with which uranium does not interfere.

THE END.

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